Method Validation for Selecting Baseline Solvent

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Outline

- Introduction & Background
- Objective
- Experimental Method
- Results & Discussion
- Conclusion

Introduction & Background

WSTF's Experience

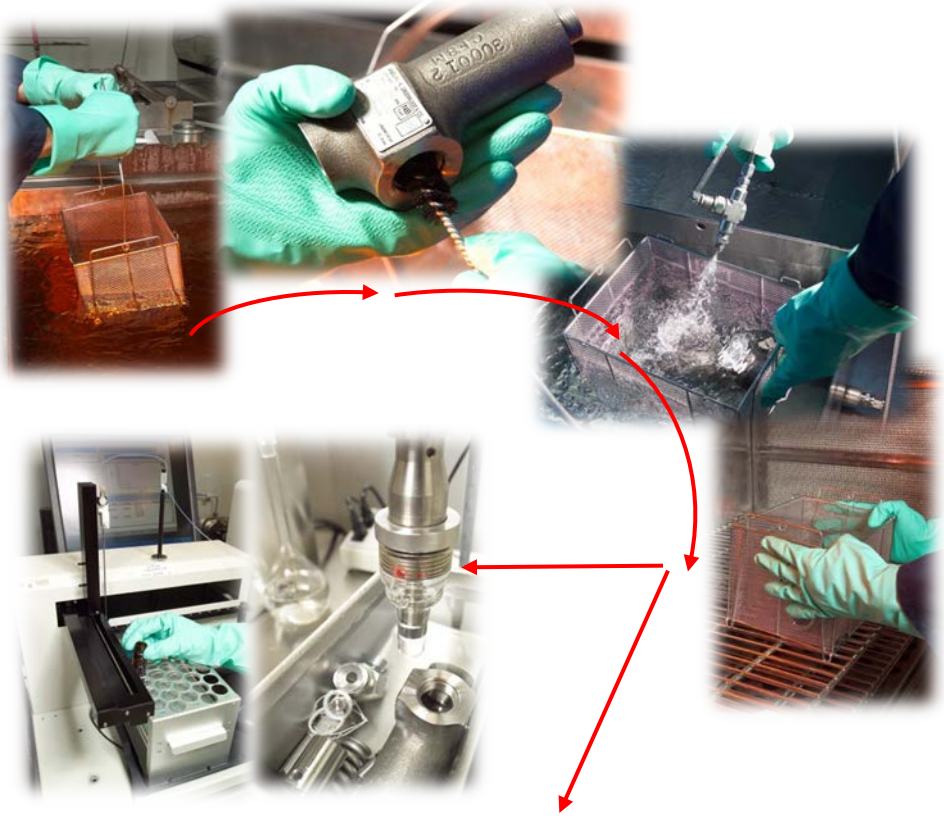
Replacing CFC-113 in the 90's

- Collaboration
  - DOD
  - NASA
    - Contractors
  - Solvent Manufactures
- Solvents
  - CFC
  - HCFC
  - HFE
  - TCE

- Considerations for Solvents
  - Cleaning effectiveness
  - O_2 compatibility
  - Materials compatibility
  - Aerospace fluids compatibility

WSTF Cleaning Process

- Pre-cleaning
  - Ultra sonic
- Visual inspection
- Final clean/ cleanliness verification
  - DI water
  - HFE 7100
  - IPA
- Process Validation
  - AK 225G (baseline solvent)



Problem Statement

- WSTF Processes is dependent on AK-225G for process validation
- AK-225G limited availability in 2015

Objective

- To establish a standardized method for the evaluation of suitable replacement *baseline* solvent AK-225G.

Experimental Method

Standards for Testing Solvents

- ASTM G121. *Standard Practice for Preparation of Contaminated Test Coupons for the Evaluation of Cleaning Agents*. ASTM International, West Conshohocken, Pennsylvania, 1998.
- ASTM G122. *Standard Test Method for Evaluating the Effectiveness for Cleaning Agents*. ASTM International, West Conshohocken, Pennsylvania, 1996 Revised 2008.
- ASTM G127. *Standard Guide for the Selection of Cleaning Agents for Oxygen Systems.* ASTM International, West Conshohocken, Pennsylvania, 1995 revised 2008.

Attributes for a Baseline Solvent

ASTM Considerations

- Toxicity
- Carcinogenicity
- Recyclability
- Waste Disposal
- Ozone Depletion
- Inertness (Flammability and combustibility)
  - Oxygen compatibility
- Availability and technical support from supplier
- Corrosivity & material compatibility
- Cost effectiveness
- Compliance with local, state, and federal regulations
- Application and use of Solvent

Other Considerations

- HAP (Hazardous Air Pollutants)
- VOC (Volatile Organic Compounds)
- ACS reagent grade chemicals or higher
- Cleaning effectiveness
- Evaporation rate

So Many Choices!

ASTM Test Method Options

- Contamination of Coupons
  - Slurry
    - Contaminants: 1 vs. mixture
    - Concentration: 1 to 100 mg/mL
  - Application
    - 1 side vs. 2 sides
    - Pipette
    - Brush
    - Spray
    - Dip
  - Dry
    - Hang vs. laying flat
- Cleaning of coupons
  - Manufacture's recommended use of solvent
  - Sonication
  - Elevated temperatures

Experiment Outline

Steps

1. Clean the coupons (standard)
2. Weigh the coupons (Tare weight)
3. Contaminate coupons
   a) Analyze slurry filter/NVR
   b) Oven dried
4. Weigh the coupon (Determine Contaminant)
5. Clean the coupon (test solvent)
   a) Analyze rinse (filter / NVR)
   b) Oven dried
6. Weigh the coupon (Residual contaminant)
7. Verify cleanliness of coupon (AK-225-G, Verification solvent)
   a) Analyze rinse (filter / NVR)
   b) Oven dried

Matrix

- Series 1
  - 1 Contamination Slurry for each solvent
  - 3 solvents tested
  - 7 coupons for each solvent
- Series 2
  - 1 contamination Slurry
  - 6 solvents
  - 1 coupon for each solvent

Contaminants

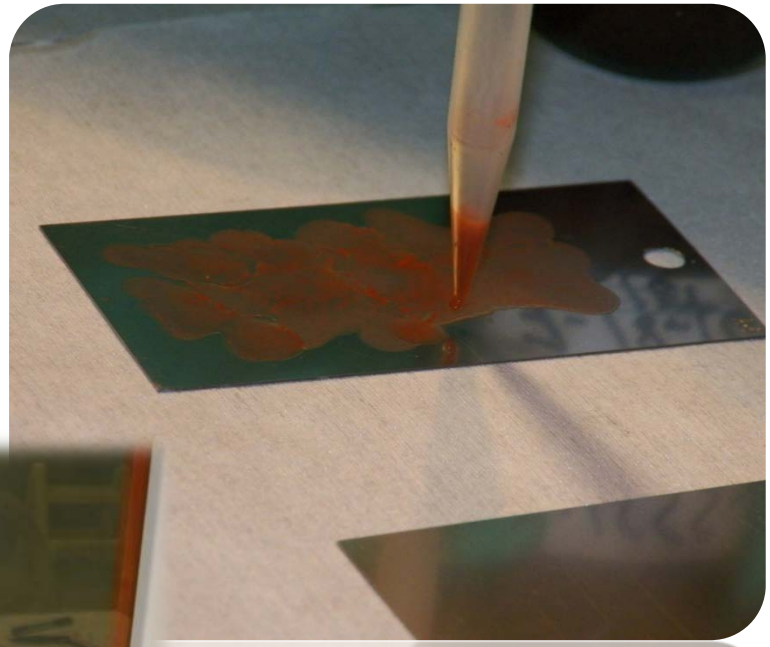
- Pump /hydrocarbon oil
- Hydraulic oil
- O_2 system lubricant
- Gage fluid
- Silicone grease
- Dye penetrates- particles
- Iron powder 60 Mesh- particles

Preparation of Contamination Slurry

- 5 slurry mixtures prepared
- 1 gram (+-.1g)
- 100 mL AK225-G
- Ultra Sonic 10 min
- Concentration 70 mg/mL

Contamination of Coupons

- 600 µL contamination Slurry
- Bake
  - 1 hr.
  - 45 ºC (+/- 5ºC)
  - Nitrogen purge
- Cool off 1 hr.
- Weighed

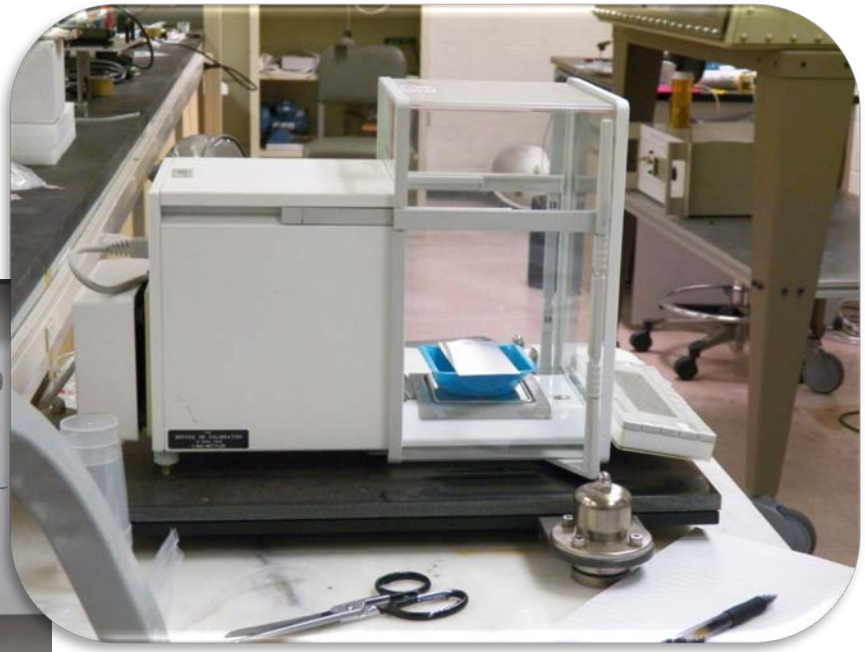


Cleaning & Verification

- Flush with 100 mL of solvent
  - NVR analysis
  - Particle count
- Cleaned with test solvent
- Verified clean with AK225G

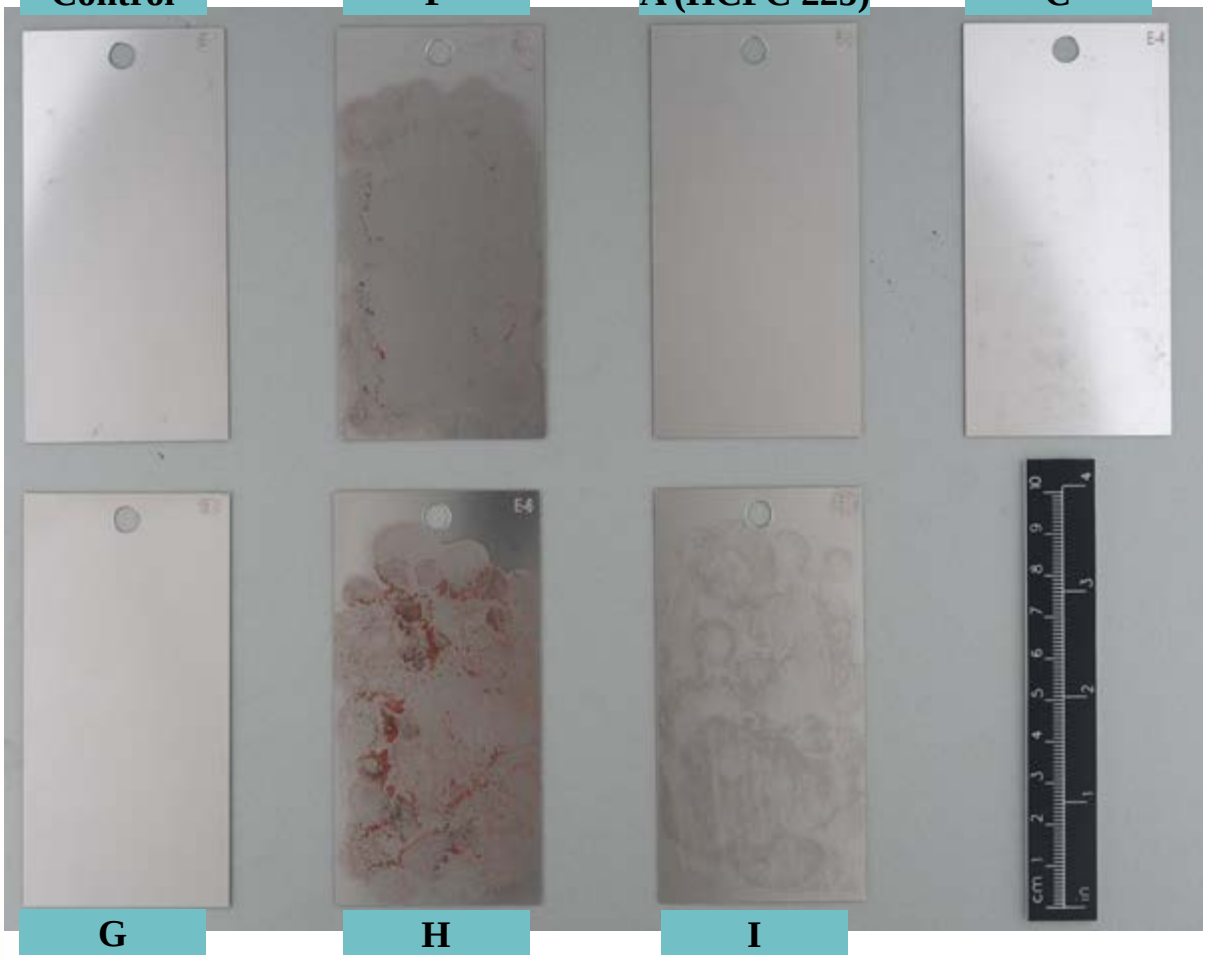
Data Collection

- Counting Particles
- Weighing coupons
- Weighing NVR
- Weighing filters



Results & Discussion

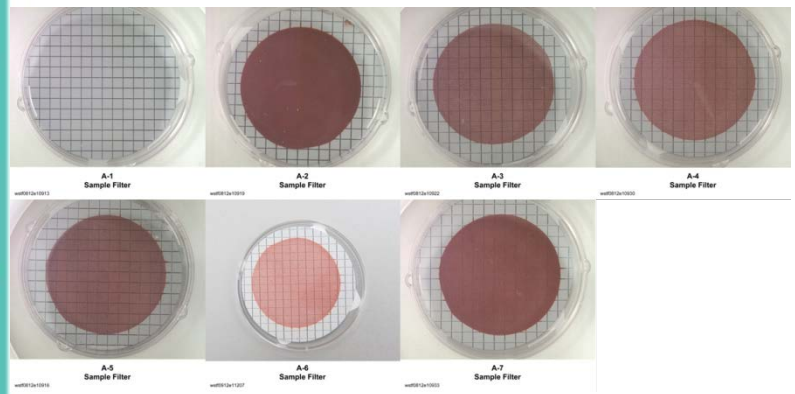
Post Cleaned, Pre AK225G Validated Coupons



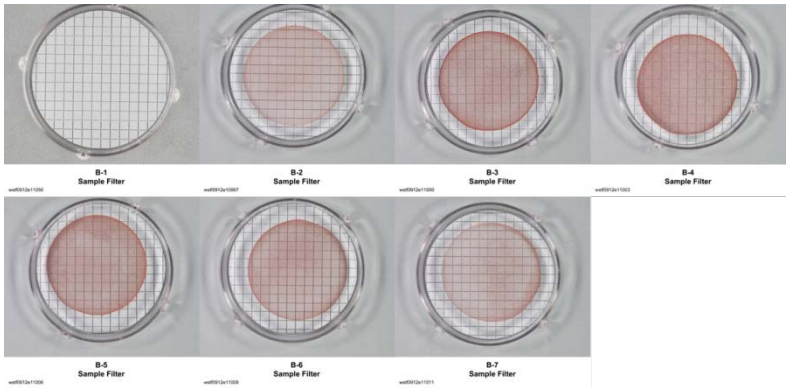
Symbol	Description	Values
General		
S	Surface Area (cm^2)	45
Ra	Surface Roughness (µm)	17
Spec. Bal	>10g-205g (g)	0.002
Clean Bal	0 - 10 mg (+-g)	0.00005
Clean Bal	> 10 mg - 1 g (+-g)	0.0001

Post Cleaned, Pre Verification Filter Papers

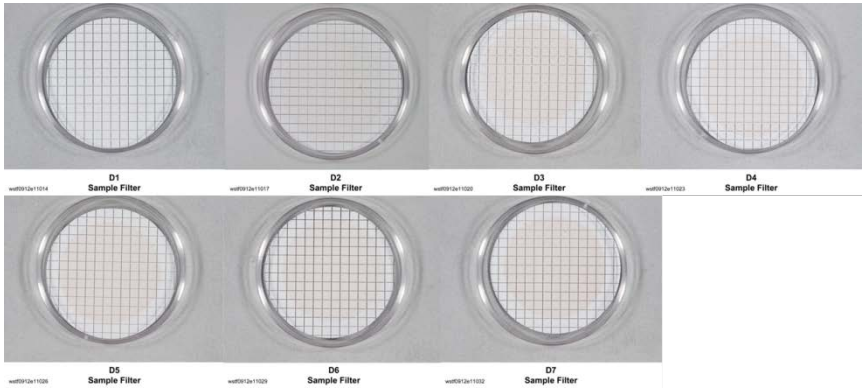
Cleaned with AK-225G



Cleaned with CFC-113

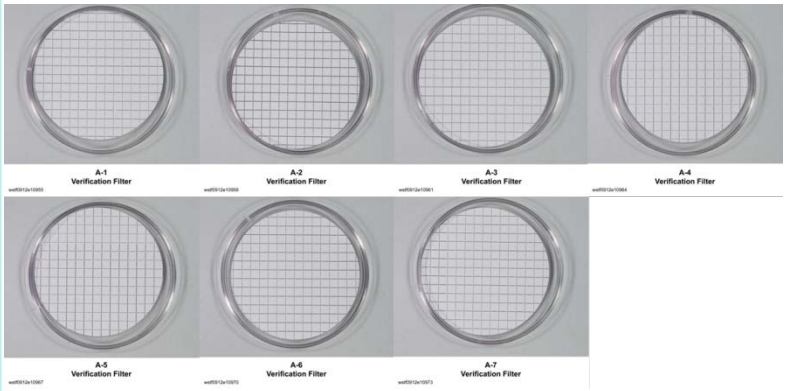


Cleaned with D

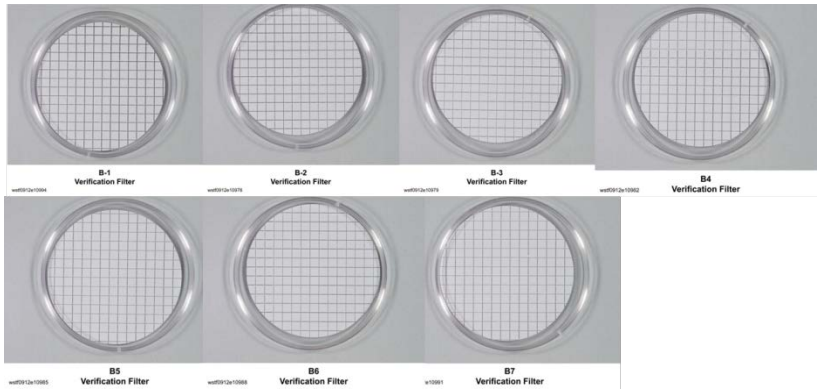


Post From AK-225G *Verification* Filter Papers

Cleaned with AK-225G



Cleaned with CFC-113



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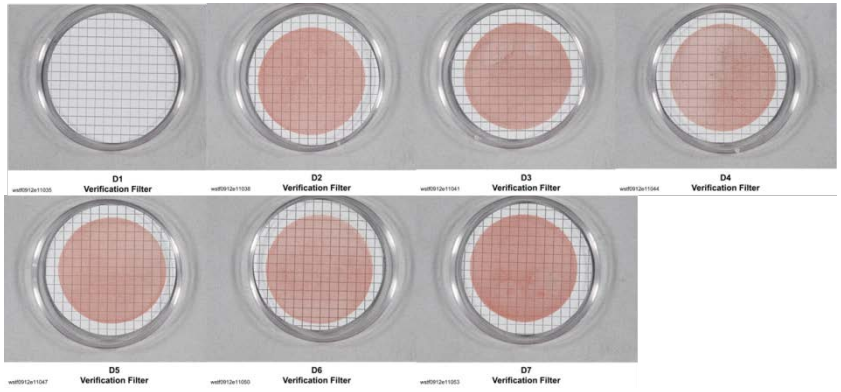

Cleaning Efficiency of Solvents

Comparison of Contamination Analyzed on Coupons

Symbol	Description	Solvents								
		*A {AK225G} (average)	*B {CFC} (average)	*D (average)	**F	**A {HCFC}	**C	**G	**H	**I
	Validation of experiment (g)	0.0007	-0.0002	0.0001	0.0004	0.0004	0.0004	0.0004	0.0004	0.0004
MCA	Mass of Contaminant Applied (g)	0.0423	0.0316	0.0359	0.0450	0.0318	0.0335	0.0334	0.0526	0.0382
MCR	Mass of Contaminant Removed (g)	0.0410	0.0312	0.0127	0.0303	0.0308	0.0323	0.0325	0.0296	0.0355
CEF	Cleaning Effectiveness Factor (%)	97.11%	98.88%	35.68%	67.33%	96.86%	96.42%	97.31%	56.27%	92.93%
C	Amount of Contamination (mg/cm^2)	0.9393	0.7015	0.7970	1.0000	0.7067	0.7444	0.7422	1.1689	0.8489
RC	Residual Contamination (mg/cm^2)	0.0270	0.0078	0.5152	0.3267	0.0222	0.0267	0.0200	0.5111	0.0600
VNVR	Verification Nonvolatile Residue Weight	0.0002	0.0005	0.0150						

Comparison of Removed Contamination Analyzed

Symbol	Description	Solvents		
		*AK-225G (average)	*CFC-113 (average)	*D (average)
SFW	Sample Filter contamination Weight (g)	0.0189	0.0054	0.7970
VFW	Verification Filter Contamination Weight (g)	0.0005	0.0000	0.5152

Key:
Baseline Solvents
* Series 1
** Series 2

Cleaning Efficiency of Solvents *Continued*

Discussion

- 3 potential baseline solvents identified
- Comparison to past test: "WSTF-IR-0134"
  - AK-225: 99% CEF
  - CFC-113: 97% CEF

Limited data on solvents, F,C,G,H,I

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Conclusion

Conclusion

- The use of AK-225G in the validation step proves additional assurance that candidate solvents qualify as baseline solvents.
- Test results that provide percent cleaning efficiencies provide guidance into selecting baseline solvents.
- Future studies should consider adequate sample size to better define cleanliness efficiency.

Questions ?

Back up slides

NVR Measured in Solvent Blanks

Solvent:	Measured NVR (mg) in 100 ml	Comments
C-HFE-(average)	3.3	Sample from Pressure Vessel
C-HFE	0.3	Sample from new bottle
D-HFE	0.4	
E-Solvent Blend	106700.3	Sample from Pressure Vessel
E-Solvent Blend	2.8	Sample from new mixture
F-HFE	0.1	Sample from bottle
G-HFE	0.2	Sample from bottle
H-HFE	0.1	Sample from bottle
I-HFE	0.3	Sample from bottle
J-Terpene	3.1	Sample from bottle.
K-HFE	0.1	Sample from bottle

- NVR criteria for solvents needs to be identified early on
- Evaporation rates role in analysis process
- Equipment compatibility with solvent
  - Beaker vs. pressure vessel

Lessons Learned/ Challenges

- Measure contaminates
- Identifying baseline, cleaning & verification solvent requirements.
- Testing location
  - Clean room
  - Vent hood
- Cross Contamination
- Experiment vs. Cleanroom practices.